



November 04, 2015

Tom Moe USS Corporation P.O. Box 417 Mountain Iron, MN 55768

RE: Project: NPDES Line 3 Wkly Pace Project No.: 1255761

Dear Tom Moe:

Enclosed are the analytical results for sample(s) received by the laboratory on October 21, 2015. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Heather R Zika

Haller Zto

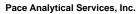
heather.zika@pacelabs.com

Project Manager

Enclosures

cc: Terri Sabetti, Northeast Technical





315 Chestnut Street Virginia, MN 55792 (218) 742-1042



CERTIFICATIONS

Project: NPDES Line 3 Wkly

Pace Project No.: 1255761

Virginia Minnesota Certification ID's

315 Chestnut Street, Virginia, MN 55792 Alaska Certification #MN01084 Arizona Department of Health Certification #AZ0785 Minnesota Dept of Health Certification #: 027-137-445

North Dakota Certification: # R-203

Wisconsin DNR Certification #: 998027470 WA Department of Ecology Lab ID# C1007 Nevada DNR #MN010842015-1

Oklahoma Department of Environmental Quality





SAMPLE SUMMARY

Project: NPDES Line 3 Wkly

Pace Project No.: 1255761

| Lab ID | Sample ID | Matrix | Date Collected | Date Received | |
|------------|--------------------------|--------|----------------|----------------|--|
| 1255761001 | WS002 Scrubber Make-up | Water | 10/21/15 08:40 | 10/21/15 13:55 | |
| 1255761002 | WS003 Thickener Overflow | Water | 10/21/15 08:40 | 10/21/15 13:55 | |



SAMPLE ANALYTE COUNT

Project: NPDES Line 3 Wkly

Pace Project No.: 1255761

| Lab ID | Sample ID | Method | Analysts | Analytes Reported | Laboratory |
|------------|--------------------------|-----------|----------|----------------------|------------|
| 1255761001 | WS002 Scrubber Make-up | EPA 200.7 | MAR | 3 | PASI-V |
| | | EPA 300.0 | DMB | 1 | PASI-V |
| 1255761002 | WS003 Thickener Overflow | EPA 200.7 | MAR | 3 | PASI-V |
| | | EPA 300.0 | DMB | 1 | PASI-V |



ANALYTICAL RESULTS

Project: NPDES Line 3 Wkly

Pace Project No.: 1255761

Date: 11/04/2015 01:33 PM

| Sample: WS002 Scrubber Make-u | ıp Lab ID: | 1255761001 | Collected | : 10/21/15 | 08:40 | Received: 10/ | 21/15 13:55 Ma | atrix: Water | |
|---|--------------------------|----------------------------------|--|------------------------------------|----------------------------|--|---|----------------------|------|
| | | | Report | | | | | | |
| Parameters | Results | Units | Limit | MDL | DF | Prepared | Analyzed | CAS No. | Qua |
| 200.7 MET ICP, Lab Filtered | Analytical | Method: EPA | 200.7 Prepar | ation Meth | od: EP/ | A 200.7 | | | |
| Calcium, Dissolved | 89.7 | mg/L | 5.0 | 0.29 | 10 | 10/23/15 11:36 | 10/27/15 15:24 | 7440-70-2 | |
| Magnesium, Dissolved | 203 | mg/L | 5.0 | 0.67 | 10 | 10/23/15 11:36 | 10/27/15 15:24 | 7439-95-4 | |
| Total Hardness, Dissolved | 1060 | mg/L | 100 | 50.0 | 10 | 10/23/15 11:36 | 10/27/15 15:24 | | |
| 300.0 IC Anions 28 Days | Analytical | Method: EPA | 300.0 | | | | | | |
| Sulfate | 744 | mg/L | 20.0 | 0.89 | 10 | | 10/30/15 13:53 | 14808-79-8 | |
| | | | | | | | | | |
| Sample: WS003 Thickener Overflo | ow Lab ID: | 1255761002 | Collected | : 10/21/15 | 08:40 | Received: 10/ | 21/15 13:55 Ma | atrix: Water | |
| Sample: WS003 Thickener Overflo | ow Lab ID: | 1255761002 | Collected Report | : 10/21/15 | 5 08:40 | Received: 10/ | 21/15 13:55 Ma | atrix: Water | |
| Sample: WS003 Thickener Overflo | ow Lab ID: Results | 1255761002 Units | | : 10/21/15 MDL | 5 08:40 DF | Received: 10/ | 21/15 13:55 Ma - Analyzed | catrix: Water | Qual |
| Parameters | Results | | Report Limit | MDL | DF | Prepared | | | Qual |
| Parameters 200.7 MET ICP, Lab Filtered | Results | Units | Report Limit | MDL | DF | Prepared | | CAS No. | Qual |
| Parameters 200.7 MET ICP, Lab Filtered Calcium, Dissolved | Results Analytical | Units Method: EPA 2 | Report Limit 200.7 Prepar | MDL ation Meth | DF nod: EP/ | Prepared A 200.7 | Analyzed | CAS No. 7440-70-2 | Qual |
| Parameters 200.7 MET ICP, Lab Filtered Calcium, Dissolved Magnesium, Dissolved | Results Analytical | Units Method: EPA 2 mg/L | Report Limit 200.7 Prepar 5.0 | MDL ation Meth | DF nod: EP/ | Prepared A 200.7 10/23/15 11:36 | Analyzed 10/27/15 15:27 | CAS No. 7440-70-2 | Qual |
| · | Analytical 889 22.3 2310 | Units Method: EPA 2 mg/L mg/L | Report Limit 200.7 Prepar 5.0 5.0 100 | MDL ration Meth 0.29 0.67 | DF nod: EP/ 10 10 | Prepared A 200.7 10/23/15 11:36 10/23/15 11:36 | Analyzed 10/27/15 15:27 10/27/15 15:27 | CAS No. 7440-70-2 | Qual |



QUALITY CONTROL DATA

Project: NPDES Line 3 Wkly

Pace Project No.: 1255761

Date: 11/04/2015 01:33 PM

QC Batch: MPRP/6057 Analysis Method: EPA 200.7

QC Batch Method: EPA 200.7 Analysis Description: 200.7 MET Dissolved

Associated Lab Samples: 1255761001, 1255761002

METHOD BLANK: 261047 Matrix: Water

Associated Lab Samples: 1255761001, 1255761002

Reporting Blank Parameter Limit MDL Result Qualifiers Units Analyzed Calcium, Dissolved ND 0.50 0.029 10/27/15 13:48 mg/L Magnesium, Dissolved mg/L ND 0.50 0.067 10/27/15 13:48

LABORATORY CONTROL SAMPLE: 261048 Spike LCS LCS % Rec Parameter Units Conc. Result % Rec Limits Qualifiers Calcium, Dissolved 50 52.1 104 85-115 mg/L Magnesium, Dissolved 50 52.0 104 85-115 mg/L

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 261049 261050 MSD MS 1255746001 Spike Spike MS MSD MS MSD % Rec Max Parameter Units Result Conc. Conc. Result Result % Rec % Rec Limits **RPD** RPD Qual Calcium, Dissolved mg/L 4.9 50 50 56.4 56.2 103 102 70-130 20 Magnesium, Dissolved mg/L 2.4 50 50 53.8 53.5 103 102 70-130 20

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 261052 261051 MS MSD MS 1255730003 MSD MS Spike Spike MSD % Rec Max Parameter Units Result Conc. Conc. Result Result % Rec % Rec Limits **RPD** RPD Qual Calcium, Dissolved 50 238 50 286 289 97 102 70-130 20 mg/L 39.3 50 Magnesium, Dissolved 50 90.1 88.9 101 99 70-130 20 mg/L 1

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.



QUALITY CONTROL DATA

Project: NPDES Line 3 Wkly

Pace Project No.: 1255761

Date: 11/04/2015 01:33 PM

QC Batch: WETA/14452 Analysis Method: EPA 300.0

QC Batch Method: EPA 300.0 Analysis Description: 300.0 IC Anions

Associated Lab Samples: 1255761001, 1255761002

METHOD BLANK: 263360 Matrix: Water

Associated Lab Samples: 1255761001, 1255761002

Parameter Units Result Limit MDL Analyzed Qualifiers

Sulfate mg/L ND 2.0 0.089 10/30/15 10:48

LABORATORY CONTROL SAMPLE: 263361

Spike LCS LCS % Rec Parameter Units Conc. Result % Rec Limits Qualifiers Sulfate mg/L 50 52.4 105 90-110

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 263362 263363

MS MSD 1256272001 Spike Spike MS MSD MS MSD % Rec Max Parameter Units Result Conc. Conc. Result Result % Rec % Rec Limits RPD RPD Qual Sulfate 50 20 mg/L 50 61.6 61.1 105 104 90-110

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 263364 263365

MS MSD MS MSD MS 1255869001 Spike Spike MSD % Rec Max Limits RPD Parameter Units Result Conc. Conc. Result Result % Rec % Rec RPD Qual Sulfate 35.2 250 250 293 292 103 103 90-110 0 20 mg/L

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.



QUALIFIERS

Project: NPDES Line 3 Wkly

Pace Project No.: 1255761

DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

LABORATORIES

Date: 11/04/2015 01:33 PM

PASI-V Pace Analytical Services - Virginia



QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: NPDES Line 3 Wkly

Pace Project No.: 1255761

Date: 11/04/2015 01:33 PM

| Lab ID | Sample ID | QC Batch Method | QC Batch | Analytical Method | Analytical Batch |
|--------------------------|--|------------------------|--------------------------|------------------------|----------------------|
| 1255761001 1255761002 | WS002 Scrubber Make-up WS003 Thickener Overflow | EPA 200.7 EPA 200.7 | MPRP/6057 MPRP/6057 | EPA 200.7 EPA 200.7 | ICP/4692 ICP/4692 |
| 1255761001 1255761002 | WS002 Scrubber Make-up WS003 Thickener Overflow | EPA 300.0 EPA 300.0 | WETA/14452 WETA/14452 | | |

Address: Company:

USS Corporation P.O. Box 417

Required Client Information:

Required Project Information:

Section C Invoice Information: Attention:

Report To: Tom Moe

Section A

CHAIN-OF-CUSTODY / Analytical Request Document
The Chain-of-Custody is a LEGAL DOCUMENT. All relevent 1.04.4.2

W0#:1255761 PM: HRZ

CLIENT: USS CORP Due Date: 11/04/15

Page 10 of 11

| | | | | | | | | 70 V 27.) 3 | | | | | | , A | | | ITEM # | | | , industria | Regueste | Phone: | Mt. Iron, I | Address: | Ī |
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| | | į | | | The second secon | | | 000 | - | | | | | | WS-003 Thickner Overflow | WS-002 Scrubber Make-Up | One Character per box. (A-Z, 0-9 / , -) Sample lds must be unique | SAMPLE ID | | a bay bary. | Reguested Due Date: | Π by: | Mt. Iron, MN 55768 | | l |
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ace Analytical "

hold, incorrect preservative, out of temp, incorrect containers)

Document Name: Sample Condition Upon Receipt Form

Document No.: F-VM-C-001-Rev.09 Document Revised: 23Feb2O15 Page 1 of 1

Issuing Authority:

Pace Virginia, Minnesota Quality Office

| Sample Condition Upon Receipt Client Name: | sal | | Project | WO#: 1255761 |
|--|-----------------|----------|-----------------------|--|
| Courier: Fed Ex . UPS | USPS | | Client | |
| Commercial Pace Tracking Number: | Other: | | · ······ · | 1255761 |
| Custody Seal on Cooler/Box Present? Yes | No. | Seals | Intact? | Yes No Optional: Proj. Due Date: Proj. Name: |
| Packing Material: Bubble Wrap Bubble B | ags 🔲 No | one [| Other:_ | Temp Blank? ✓ Yes ☐ No |
| Thermometer Used: 140792808 | Type of I | ce: 💆 | Wet [| Blue None Samples on ice, cooling process has begun |
| Cooler Temp Read °C: 1.2 Cooler Temp Temp should be above freezing to 6°C Correction Fac | Corrected *C | : L | | Biological Tissue Frozen? Yes No NA d Initials of Person Examining Contents: Comments: |
| Chain of Custody Present? | Yes | □No | □N/A | 1. |
| Chain of Custody Filled Out? | Yes | □No | □N/A | 2. |
| Chain of Custody Relinquished? | Yes | □No | N/A | 3. |
| Sampler Name and Signature on COC? | Yes | □No | □N/A | 4. |
| Samples Arrived within Hold Time? | Yes | □No | □N/A | 5. |
| Short Hold Time Analysis (<72 hr)? | ∏Yes | _[]WO | □N/A | 6. |
| Rush Turn Around Time Requested? | □Yes | D#10 | □N/A | 7. |
| Sufficient Volume? | Tes. | □No | □N/A | 8. |
| Correct Containers Used? | Yes | □No | □n/a | 9. |
| -Pace Containers Used? | Yes | □No | □N/A | |
| Containers Intact? | Hes | □No | □n/a | 10. |
| Filtered Volume Received for Dissolved Tests? | Yes | .DNO | N/A | 11. Note if sediment is visible in the dissolved containers. |
| Sample Labels Match COC? -Includes Date/Time/ID/Analysis Matrix: | Yes | □No | □N/A | 12. |
| All containers needing acid/base preservation will be checked and documented in the pH logbook. | □Yes | □No | ZIN/A | See pH log for results and additional preservation documentation |
| Headspace in Methyl Mercury Container | Yes | □No | _DN/A | 13. |
| Headspace in VOA Vials (>6mm)? | Yes | □No | ;EN/A | 14. |
| Trip Blank Present? | Yes | □No | N/A | 15. |
| Trip Blank Custody Seals Present? | ∏Yes | □No | ₽N/A | |
| Pace Trip Blank Lot # (if purchased): | | | | |
| CLIENT NOTIFICATION/RESOLUTION | | | | Field Data Required? Yes No |
| Person Contacted: | | | . [| Date/Time: |
| Comments/Resolution: | | | | |
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| FECAL WAIVER ON FILE Y N | ,, | TEM | PERATUI | RE WAIVER ON FILE Y N |
| Project Manager Review: Hotel Whenever there is a discrepancy affecting North Carolin hold incorrect prospective out of town incorrect prospective out of the prospective out of town incorrect prospective out of the prospective out | na compliance : | samples, | a copy of th | Date: 10 00 115 is form will be sent to the North Carolina DEHNR Certification Office (i.e. out of |